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IS 12308-7 (1991): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 7: Determination of nickel by dimethyl glyoxime (gravimetric) method(for nickel 0.5 to 36 percent) [MTD 7: Light Metals and their Alloys]

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ढलवां लोहे एवं कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियाँ

भाग 7 डाइमिथाइल ग्लाइअक्सिम (भारात्मक) पद्धति से निकिल ज्ञात करना (0·5 से 36 प्रतिशत निकिल के लिए)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 7 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL 0·5 TO 36 PERCENT)

UDC 669·162·272·12+669·13 : 543·21 [546·74]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1991 Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 7) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of Chemical analysis of pig iron, cast iron and plain carbon and low alloy steels *(revised)*.' During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly, IS 228 on revision was published in several parts covering chemical analysis of various steels only and separate series of standard under IS 12308 is being published for chemical analysis of cast iron and pig iron. This standard (Part 7) is one in the latter series. The other parts in the series are as follows:

Part 1 Determination of total carbon by thermal conductivity method

Part 2 Determination of sulphur by iodimetric titration method

Part 3 Determination of manganese by periodate spectrophotometric method

Part 4 Determination of total carbon, graphitic carbon and combined carbon by gravimetric method

Part 5 Determination of phosphorus by alkalimetric method (for phosphorus 0·01 to 0·50 percent)

Part 6 Determination of silicon (for silicon 0·1 to 6·0 percent)

Part 8 Determination of chromium by persulphate oxidation method (for chromium 0·1 to 28 percent)

Part 9 Determination of molybdenum by thiocyanate (spectrophotometric) method (For molybdenum 0·1 to 1·0 percent)

Part 10 Determination of manganese (up to 7·0 percent) by arsenite volumetric method

Part 11 Determination of total carbon by the direct combustion (volumetric) method

Part 12 Determination of total carbon by the direct combustion volumetric method (for carbon 15·0 to 4·50 percent)

In this part dimethyl-glyoxime (gravimetric) method for determination of nickel in cast iron and pig iron has been prescribed on the basis of experience gained during the past and reproducibility of the method has been incorporated at different percent levels of nickel.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values *(revised)*'.

Indian Standard
METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 7 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD (FOR NICKEL 0·5 TO 36 PERCENT)

1 SCOPE

This standard (*P*art 7) covers the method for determination of nickel in the range from 0·5 to 36 percent in alloy cast iron and pig iron.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard.

IS No.	*Title*
264 : 1976	Nitric acid *(second revision)*
265 : 1987	Hydrochloric acid *(third revision)*

3 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant Indian Standard.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF NICKEL BY DIMETHYL-GLYOXIME (GRAVIMETRIC) METHOD

5.1 Outline of the Method

After separating silicon, nickel is precipitated as nickel dimethyl-glyoximate and weighed.

5.2 Reagents

5.2.1 *Concentrated Nitric Acid*, rd = 1·42 (conforming to IS 264 : 1976).

5.2.2 *Dilute Hydrochloric Acid*, 1 : 1 (*v*/*v*).

5.2.3 *Dilute Sulphuric Acid*, 1 : 1 (*v*/*v*).

5.2.4 *Hydrofluoric Acid*, 40 percent.

5.2.5 *Potassium Bisulphate*, solid.

5.2.6 *Potassium Pyrosulphate*, solid.

5.2.7 *Citric Acid Solution*, 50 percent (*m*/*v*).

5.2.8 *Concentrated Ammonia Solution*, rd = 0·90.

5.2.9 *Dimethyl-glyoxime Solution* (1 percent).

Dissolve 1 g of dimethy-glyoxime in 80 ml of rectified spirit and before use make up to 100 ml with the spirit. Filter before use.

5.3 Procedure

5.3.1 Transfer an appropriate quantity of the sample that will contain 20 to 60 mg of nickel, to a 400 ml beaker. Add 30-40 ml of dilute hydrochloric acid (1 : 1). Heat gently and when the reaction has subsided, add 5 ml concentrated nitric acid to oxidise the solution. Evaporate the solution to dryness and bake for 30 minutes at 120°C. Add to it 25-30 ml of dilute hydrochloric acid (1 : 1) and heat for dissolution. Filter through a medium textured filter paper and wash with hot water. Preserve the filtrate.

5.3.2 Ignite the residue in a platinum crucible at 800°C. Cool, moisten with a few drops of dilute sulphuric acid (1 : 1) and add 5-10 ml hydrofluoric acid. Evaporate to dryness and fuse the residue with potassium bisulphate, potassium pyrosulphate and take the mass in dilute hydrochloric acid (1 : 1) and mix the solution with the filtrate reserved under **5.3.1**.

5.3.3 Dilute the solution to about 400 ml with water and add 50 ml of citric acid solution. Neutralise with ammonia solution and acidify with dilute hydrocholric acid (1 : 1). Heat the solution to 90°C and add excess of dimethyl-glyoxime solution (10 ml for every 10 mg of Nickel). Neutralise the solution with ammonia solution and add 2-3 ml excess and stir well. Keep the solution at about 60°C for one hour and then cool to room temperature, filter and wash 8-10 times with cold water containing few drops of ammonia solution.

NOTE — As copper, cobalt and manganese also consume dimethy-glyoxime, additional excess of another 25-30 ml of dimethyl-glyoxime solution must be added, if any are present.

5.3.4 Dissolve the precipitate by adding hot dilute hydrochloric acid (1 : 1) on the filter paper and collect the solution in the same beaker in which precipitation was carried out. Cool and dilute to 300 ml. Add 10 ml of citric acid solution, neutralize the solution with ammonia solution and acidify again with dilute hydrochloric acid (1 : 1). Heat the solution to 90°C and add dimethyl-glyoxime solution in excess, in the proportion 10 ml for each 10 mg of Nickel expected. Neutralise the solution with ammonia solution and add 2 ml excess. Keep the solution at about 60°C for one hour. Cool the solution and filter through a sintered glass crucible (No. 3) which is previously dried at 120°C and weighed to constant mass (M_1). Wash the precipitate 10-15 times with water containing few drops of ammonia solution. Dry the precipitate at 150°C to constant mass. Cool in a desiccator and weigh (M_2).

5.4 Calculation

$$\text{Nickel, percent by mass} = \frac{A}{B} \times 20{\cdot}32$$

where

$A = (M_2 - M_1) =$ mass, in g, of nickel dimethyl-glyoxime; and

$B =$ mass, in g, of sample taken.

5.5 Reproducibility

± 0·025 at 0·5 percent nickel,

± 0·03 at 1 percent nickel,

± 0·07 at 5 percent nickel,

± 0·12 at 10 percent nickel,

± 0·17 at 15 percent nickel,

± 0·22 at 20 percent nickel,

± 0·28 at 25 percent nickel,

± 0·33 at 30 percent nickel.

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